IS 5343 (1996): Butylated Hydroxyanisole, Food Grade [FAD 8: Food Additives]

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(Reaffirmed 2001)

भारतीय मानक

बूटाईलेटेड हाईड्रॉक्सीएनीसोल, खाद्य ग्रेड — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

BUTYLATED HYDROXYANISOLE, FOOD GRADE — SPECIFICATION

(First Revision)

ICS 67.220.20

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1996

Price Group 3

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve the appearance, flavour, texture or storage properties, and in some cases to enhance the nutritive value of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards was, therefore, prepared by this Bureau to cover purity and identification of these substances. It is hoped that these standards would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and international bodies and the consumer industries to use it within the limits permitted by the Health authority.

Butylated hydroxyanisole (BHA), food grade is used as an antioxidant. BHA is permitted for use in foods under *The Prevention of Food Adulteration Rules,* 1955. These rules, *inter-alia* prescribe:

'The food additives permitted for use in certain foods shall be sold only under the BIS Certification Mark'. Butylated Hydroxyanisole, food grade is one such item.

This standard was first published in 1969. It is being revised to include the following changes/additions:

a) To provide a separate clause for description incorporating the solubility properties to keep the standard in line with the Food Chemical Codex, NRC.

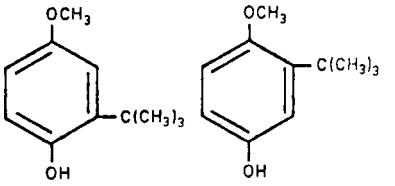
b) To provide additional requirements for 3-tertiary-butyl 4-hydroxyanisole, phenolic impurities and specific absorption and their test methods.

c) To replace lead by heavy metals and the corresponding test method.

d) To provide for marking, directions for storage and expiry date under the marking clause.

Chemical Names and Formulae

A mixture of 3- and 2-tertiary butyl-4-hydroxyanisole; a mixture of 3- and 2-tertiary butyl-4-methoxyphenol. Its empirical formula is $C_{11}H_{16}O_2$ and molecular weight is 180.24. Structural formula of BHA is:



In the preparation of this standard considerable amount of assistance has been derived from the following :

Specification for identity and purity of food additives. Vol I Antimicrobial preservatives and antioxidants 1962. Food and Agricultural Organization of the United Nations, and the World Health Organization, Rome.

Food chemical codex, Third Edition, National Academy of Sciences, National Research Council, Washington DC.

(*Continued on third cover page*)

Indian Standard

BUTYLATED HYDROXYANISOLE, FOOD GRADE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of test for butylated hydroxyanisole (BHA), food grade.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1699 : 1995	Methods of sampling and test for synthetic food colours (*second revision*)
5058 : 1996	Sodium citrate, food grade (*first revision*)

3 DESCRIPTION

BHA is a mixture of 3- and 2-isomer. It is white or slightly yellow waxy crystalline solid with an aromatic odour. The material is insoluble in water, freely soluble in ethanol and propylene glycol.

NOTE — The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity and dependence must be placed on other specifications.

4 REQUIREMENTS

4.1 Identification

4.1.1 When 2 ml of 2.0 percent aqueous borax solution and a few small crystals of 2, 6-dichloroquinonechlorimide are added to an ethanolic solution [1 percent (*m/v*)] of butylated hydroxyanisole, a blue colour shall appear.

4.1.2 When 2 ml of ferric chloride (0.2 percent, $FeCl_3.6H_2O$ in absolute ethanol) and 2 ml of 0.2 percent 2,2' bipyridine in absolute ethanol are added to 5 ml of 0.5 percent butylated hydroxyanisole in 50 percent ethanol, a red colour shall appear.

4.2 The material shall also conform to the requirements given in Table 1.

5 PACKING, STORAGE AND MARKING

5.1 Packing

The material shall be securely packed in well-filled containers with minimum access to light and air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

5.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

5.3 Marking

Each container shall be legibly and indelibly marked with the following information:

a) Name of the material, including the words 'Food Grade';

b) Name and address of the manufacturer;

c) Net content, when packed;

d) Batch or code number;

e) Directions for storage;

f) Expiry/Best before date; and

g) Any other requirements as specified under the *Standards of Weights and Measures (Packaged Commodities) Rules,* 1977 *Prevention of Food Adulteration Rules,* 1955.

5.3.1 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.3.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Table 1 Requirements for BHA, Food Grade

(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Other Standards.
(1)	(2)	(3)	(4)	(5)
i)	a) Purity as $C_{11}H_{16}O_2$, percent by mass, *Min*	98.5	A	—
	b) 3 tertiary butyl 4-hydroxyanisole, percent by mass, *Min*	85	A-1.1.2 & A-1.1.2.1	—
ii)	Melting point °C	48 to 63	B	—
iii)	Sulphated ash; percent by mass, *Max*	0.05	C	—
iv)	Arsenic (as As), mg/kg, *Max*	3	—	15 of IS 1699:1995
v)	Heavy metals (as Pb), mg/kg, *Max*	10	—	Annex E of IS 5058:1996
vi)	Iron (as Fe), mg/kg, *Max*	5	D	—
vii)	Phenolic impurities, percent by mass, *Max*	0.5	E	—
viii)	Specific absorption E 1 percent (1 cm cell) in ethanol at			—
	a) 290 nm	190 *Min* 210 *Max*	F	
	b) 228 nm	326 *Min* 345 *Max*	F	

6 SAMPLING

The representative samples of the material shall be drawn according to the method prescribed in 4 of IS 1699 : 1995.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF PURITY

A-0 GENERAL

Two methods, that is, infrared and colorimetric methods, have been specified. Either method could be used.

A-1 INFRARED METHOD

A-1.1 Procedure

Weigh 1 000 g of butylated hydroxyanisole into a 10-ml volumetric flask, dissolve it in carbon disulphide, dilute to the mark with this solvent and mix thoroughly. Fill a 0.15 mm liquid cell with the solution, insert in an infrared spectrometer and measure the spectrum from 10.5 to 12.5 μ using the 1.3 cm rock salt plate in the reference beam, 2x slits and normal scanning speed. Draw a background line on the spectrogram from 11.0 to 12.0 μ. Determine the net absorption of the sample at 11.42 μ by subtracting the background absorption at this wave length from the total absorption of the

sample. Refer the net absorption value at a previously prepared standard reference curve to obtain the apparent butylated hydroxyanisole assay.

A-1.1.1 *Preparation of Standard Reference Curve*

Weigh 0.900, 0.950 and 1.000 g of 3-tertiarybutylhydroxyanisole reference standard into three 10-ml volumetric flasks. Dissolve the samples in carbon disulphide, dilute to the mark with this solvent and mix thoroughly. Measure the spectra of these three samples using the same conditions described under **A-1.1**, obtain the net absorption of the three samples at 11.42 μ and plot these values against the percentage of butylated hydroxyanisole. The 0.900, 0.950 and 1.000 g samples represent 90, 95 and 100 percent butylated hydroxyanisole, respectively.

A-1.1.2 *Isomer Ratio Test*

Melt the sample in a water-bath and stir thoroughly. Weigh 1.000 g of the molten sample into a 10-ml volumetric flask. Dilute to the mark with carbon disulphide and shake until the sample is completely dissolved. Measure the infrared spectrum of the solution from 10 to 12 μ using a 0.4-mm cell with a 1.3-cm rock salt plate in the reference beam. From the percentage transmittance readings at 10.75 and 10.95 μ, calculate the optical density values. Divide the optical density at 10.75 μ by that at 10.95 μ to obtain the optical density ratio. (Exact position of these absorption bands may vary, depending upon the instrument. If a recording instrument is used, the position of minimum transmission on the chart should be taken with a nonrecording instrument, the exact length and slit setting should be determined.) Using the optical density ratio value, determine the percentage of 3-tertiarybutylhydroxyanisole in the sample by means of a calibration curve.

A-1.1.2.1 *Preparation of calibration curve*

Weigh (a) 1 000 mg of 3-tertiarybutylhydroxyanisole; (b) 900 mg of 3-tertiarybutylhydroxyanisole and 100 mg of 2-tertiarybutylhydroxyanisole; (c) 800 mg of 3-tertiarybutylhydroxyanisole and 200 mg of 2-tertiarybutylhydroxyanisole to an accuracy of ±1 mg into three 10-ml volumetric flasks, dilute to the mark with carbon disulphide and shake until the sample is completely dissolved. Measure the infrared spectra for each of these three mixtures following the same procedure used for the sample. Plot the calculated optical density ratios obtained against the corresponding concentrations of 3-tertiarybutylhydroxyanisole.

A-1.2 Calculation

Calculate the true butylated hydroxyanisole assay using the following equation:

Butylated hydroxyanisole, percent by mass = Apparent butylated hydroxyanisole assay (**A-1.1** and **A-1.1.1**) + 0.16 [100 - percent of 3-tertiarybutylhydroxyanisole (**A-1.1.2** and **A-1.1.2.1**)].

A-2 COLORIMETRIC METHOD

A-2.1 Reagents

A-2.1.1 *Ethanol* — 80 percent.

A-2.1.2 *Borax* — 2.0 percent, aqueous.

A-2.1.3 *2,6-dichloroquinonechlorimide* — 0.01 percent.

A-2.2 Procedure

Prepare a solution of pure butylated hydroxyanisole in 80 percent ethanol containing 5.0 μg per millilitre. Place suitable aliquots (1 to 12 ml) of the butylated hydroxyanisole solution into small glass-stoppered bottles to give a range of 5-60 μg per aliquot. Add enough 80 percent ethanol to each bottle to give a total of 12 ml. Then add 2 ml of aqueous borax and 2 ml of 2,6-dichloroquinonechlorimide. Age the samples and the blank for 15 minutes. Using the blank as a reference standard, determine the optical density at 610 nm on a colorimeter or spectophotometer. Plot the standard curve on regular coordinate paper using optical density versus concentration of butylated hydroxyanisole per aliquot. The points should fall on or near a straight line.

Proceed as above using a sample solution in 80 percent ethanol. From the optical density find out the purity of butylated hydroxyanisole using the standard curve.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF MELTING POINT

B-1 APPARATUS

B-1.1 Oven or Oil-Bath

Maintained at about 75°C.

B-1.2 Sample Tube

25 mm × 150 mm test-tube closed with a cork stopper having two holes – one at the centre to take thermometer and one at the side to take an agitator.

B-1.3 Agitator

With a paddle formed by bending a piece of stainless steel wire to form a loop surrounding the thermometer.

B-1.4 Thermometer

B-1.5 Air Bath Tube

B-1.6 Water-Bath

Maintained between 55° and 60°C.

B-2 PROCEDURE

Melt a representative sample by means of an oven or oilbath at about 75°C. Take a sample tube fill it to a depth of about 90 mm. Insert the stopper carrying the thermometer and stirrer, adjusting the thermometer so that the thermometer immersion mark is at the surface. The tip of the bulb should be about 1 cm from the bottom of the tube. Place the sample tube in an air-bath tube, and then place the air-bath tube in water-bath maintained between 55° and 60°C. Gently stir the molten sample at the rate of about 20 strokes per minute. Record temperature readings at 30 second intervals to 0.1°C. The temperature of the sample will fall gradually at first, rise slightly and become nearly constant for 3 to 5 minutes. If the lowest descending temperature is more than 1.0°C below the average temperature of the plateau, the determination should be repeated using a slightly warmer water-bath. The temperature at which the thermometer reading is constant for 5 consecutive readings is taken as the melting point.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF SULPHATED ASH

C-1 REAGENT

C-1.1 Concentrated Sulphuric Acid

C-2 PROCEDURE

Weigh accurately about 2 g of the material in a tared crucible. Ignite, gently at first, until the material is thoroughly charred, cool, moisten the residue with 1 ml of sulphuric acid and ignite gently till the carbon is completely consumed. Cool the crucible in a desiccator and weigh.

NOTE — Carry out the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of carbon.

C-3 CALCULATION

Sulphated ash, percent by mass $= \frac{M_1}{M_2} \times 100$

where

M_1 = mass in g, of the residue; and

M_2 = mass in g, of the material taken for the test.

ANNEX D

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF IRON

D-1 REAGENT

D-1.1 Bromine Solution

Prepare a saturated solution of bromine by agitating 2 to 3 ml of bromine with 100 ml of cold water in a glass-stoppered bottle, the stopper of which should be lubricated with petroleum. Store in a cool place and protect from light.

D-1.2 Hydrochloric Acid

D-1.3 Ammonium Persulphate

D-1.4 Ammonium Thiocyanate Solution

Dissolve 8 g of ammonium thiocyanate (NH_4CNS), in sufficient water to make 100 ml.

D-1.5 Standard Iron Solution

5 µg iron per ml.

D-2 PROCEDURE

To one gram of the sample add 2 ml of hydrochloric acid and evaporate to dryness on a steam-bath. Dissolve the residue in 2 ml of hydrochloric acid and 20 ml of water, add a few drops of bromine solution, and boil the solution to remove the bromine. Cool, dilute with water to 25 ml, then add 59 mg of ammonium persulphate and 5 ml of ammonium thiocyanate solution. Any red or pink colour shall not exceed that produced in a control containing 1.0 ml of standard iron solution (5 µg. Fe).

ANNEX E

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF PHENOLIC IMPURITIES

E-1 PROCEDURE

Phenolic Impurities are determined by the method using silica gel G plates.

Solution 1 — Dissolve 0.25 g of BHA in 10 ml of ether

Solution 2 — Dilute 1 ml of Solution 1 to 10 ml with ether and then dilute 1 ml of this solution to 20 ml with ether. Use the final dilution as Solution 2.

Spot 2 ml each of Solution 1 and Solution 2 on separate TLC plates and properly identify them. Place them in developing chamber containing chloroform as solvent and allow the solvent to ascend to a point of 15 cm above the sample spots. Develop the chromatograms by spraying a mixture containing 100 ml of 10.5 percent ferric ferro cyanide solution and 25 ml of 5 percent ferric chloride solution. Any blue violet spots appearing on chromatogram 1 (other than the major spot and the spot at Rf 0.35) are not more intense than the major spot appearing on chromatogram 2.

ANNEX F

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF SPECIFIC ABSORPTION

F-1 PROCEDURE

Prepare 1 percent solution of butylated hydroxyanisole in ethanol and find out its specific absorption in a suitable spectrophotometer using 1 cm cell at wavelengths 290 nm and 228 nm.

(*Continued from second cover page*)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2:1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act. 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

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(First Revision)

[*Page* 2, *Table* 1, *Sl No.* (vi)] — Delete the parameter and its requirement and renumber the subsequent Sl Nos.

(FAD 8)

Reprography Unit, BIS, New Delhi, India